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Effects of the Characteristics of High Range Water Reducing Agents and the Water to Powder Ratio on Rheological and Setting Behavior of Self-Consolidating Concrete

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ABSTRACT

To design robust self-consolidating concrete (SCC) for various environmental conditions, it is essential to understand the relevant mechanisms that control the flow performance. This paper depicts how high range water reducing agents (HRWRAs) interact with clinker and hydration phases, and it discusses the important role of the charge density of polycarboxylic HRWRAs in the way the rheology is affected. Based on the rheometric investigations on SCC mixture compositions with different water to powder ratios (w/p) and observations of their pastes' Vicat setting times, the study shows that increasing charge densities of the HRWRA and decreasing w/p reduce the flow retention and have lesser retarding effect on the setting. Based on the test results and discussions optimization procedures for the mixture composition and the HRWRA modification are suggested to achieve optimized performance for varying environmental situations and highest robustness for specific conditions.

Keywords

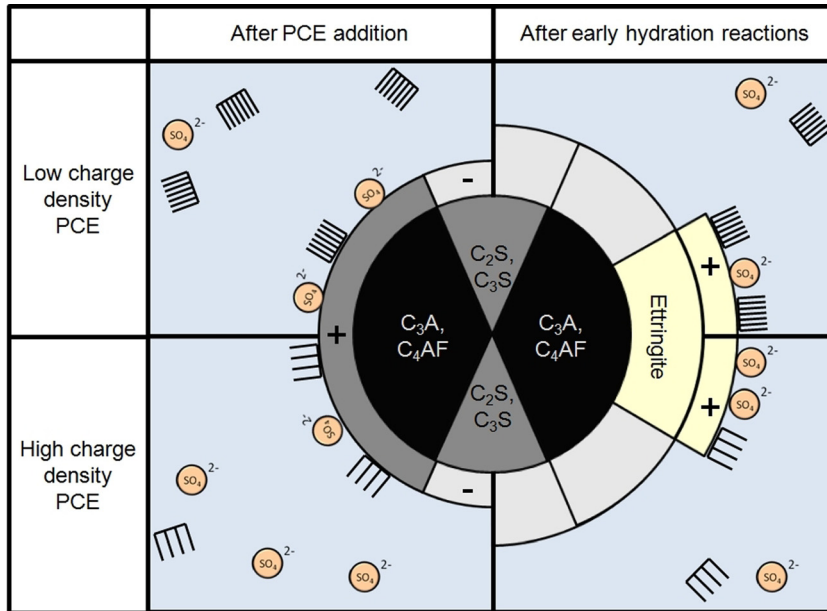
polycarboxylate, HRWRA, robustness, rheology, self-consolidating concrete

Introduction

The flow properties of self-consolidating concrete (SCC) are determined by a range of factors, such as the type, quantity, and composition of the aggregate, the water/solid particle ratio of the binding agent paste, and the interactions between the binding agent components and polymer admixtures. The latter issue is the most distinctive feature between SCC and normal concrete since high amounts of high range water reducing agents (HRWRAs) are essential for the proper flow of SCC. Due to their strong effect on the reduction of the yield stress, usually, polycarboxylate ether based HRWRA (PCE) is incorporated into SCC mixture compositions. The PCE molecules are composed of a negatively charged methacrylic main chain and a number of ethylene oxide grafts. Due to the anionic charges in the main chain, the polymers are adsorbed on positively charged surfaces, namely on aluminate and ferrous clinker phases as well as on monosulfate and ettringite hydration phases [1,2]. The actual dispersing effect, however, is caused by steric repulsion of the particles through the side chains and the main chain [3].

The addition of HRWRAs significantly affects the yield stress of a cementitious system, which can be characterized by changes of the slump flow behavior. The lower the yield stress, the larger the slump flow diameter. The quantity of adsorbed HRWRA molecules correlates linearly with the resulting slump flow [4,5]. This correlation holds true independent of the polymer geometry [6]. HRWRA molecules, which cannot be adsorbed, cannot contribute to the dispersing effect. PCE competes for adsorption sites with anions in the pore solution. Particularly, sulfate ions from the set retarder and alkali sulfates from the clinker also strive to adsorb on positively charged surface exposed areas [7,8]. This effect is shown in **Fig. 1**. The higher the polymer's anionic charge density may be, the more efficiently it can be adsorbed; the lower it is, the greater the delay in adsorption. The charge density, therefore, is the determining parameter for a PCE in terms of its efficiency to reduce the yield stress at a certain time.

If very high dosages of HRWRA are applied in SCC, significant quantities of PCE may not be adsorbed immediately because not enough adsorption sites are available. Adsorption can take place over a longer period of hydration time as additional new adsorption sites grow; thus, affecting the time dependent flow properties of the material. Both the solubility of the ions as well as the adsorption and desorption processes are dependent on time and boundary conditions such as the environmental temperature or the mixing intensity. The reaction products generated during initial and early hydration yield new surfaces for the adsorption of HRWRAs. Ettringite, in particular, exhibits a high positive zeta potential; thus offering large adsorption sites with time [2]. The time effect of the ettringite formation on the adsorption of PCE with different charge densities is also illustrated schematically in **Fig. 1**. At an early stage, highly charged PCEs can be adsorbed in large quantities, whilst low charge density PCEs are partially repulsed or desorbed by the anions

FIG. 1 Influence of charge density and hydration on the adsorption of PCE.

found in the solution. These non-adsorbed PCEs remain in the pore solution and do not contribute to dispersion. During the induction period of the cementitious hydration steady hydration takes place at relatively slow velocity, causing a constant growth in the specific surface of cement particles [9]. Over the course of time, the resulting morphological changes make the adsorbed PCEs lose their effectiveness. Low charge density PCEs are initially adsorbed at lower rate. The larger amount of non-adsorbed polymers can be adsorbed onto newly created hydration products over a delayed period of time, and thereby contribute to a long retention of the plasticizing effect.

Consequently, highly charged PCEs are well suited for the field of precast components, which demand for a rapid dispersion effect, and where any retention of the flowability after finishing the casting process is, in fact, rather undesirable. PCEs with a lower charge density are suited to uses in the ready-mix concrete sector. These are typically not quite as effective in plasticizing as highly charged polymers, but can nonetheless retain the consistency over the required period of time.

If a particular flow property is stipulated at a defined point in time, a high charge density PCE will be able to attain this with lower solid particle contents than a low charge density PCE. In a simplified way, one can imagine that a certain total charge quantity has to be adsorbed in order to generate charge equilibrium at a defined point in time. With a higher charge density, as a consequence, fewer polymers are required to attain this equilibrium. Due to effects over time, competitive adsorption, and also the effect of adsorbed PCEs on the growth of hydrates, this simplification does not fully render the complex situation but it helps to qualitatively illustrate the influence of the PCE charge density on the required amount of polymers to achieve a stipulated yield stress reduction.

TABLE 1

Clinker phase composition of the OPC based on the modified Bogue calculation after Taylor [11].

Clinker Phase	Modified Bogue Calculation [11]
C ₃ S	61.8 %
C ₂ S	20.5 %
C ₃ A	6.2 %
C ₄ AF	7.7 %
Sum:	96.1 %

Today, a wide range of different mixture composition for SCC has established. Hence, the flow properties of SCC are not only dependent on the charge density of the HRWRA but also on the total volume of paste components, as well as the water to powder ratio (w/p). SCCs that are rich of powder typically operate at a low w/p to avoid segregation. They can be characterized by low yield stress and high viscosity. SCCs designed with lower powder contents typically require higher w/p values in order to increase the total paste volume. The w/p, however, can have a supplementary strong influence on the rheology of SCC as well as on the retention of the workability. Very often, SCCs need to be amended by supplementary stabilizing agents and are featured with a relatively higher yield stress and at the same time relatively lower plastic viscosity.

In order to establish a robust casting process fitting in the particular specifications of a construction site, it is of utmost importance to be aware of the parameters that control the rheology of SCC in the course of time. Based on experiments with two characteristically different SCC types, varied PCE charge densities, and various paste compositions, the present study illustrates and explains the interacting effects determined by the w/p and the PCE modifications.

Experimental Work

Two characteristic SCC mixtures were designed, one of which is rich of powder and can be classified as powder type SCC (POW). The other concrete has significantly lower w/p and can be classified as stabilizing agent type SCC (STA) according to the Japanese regulations [10]. The pastes were generated from an ordinary Portland cement (OPC) (Tables 1 and 2) and a limestone filler with 90 % of the grains passing the 125 μm (0.005 in.) sieve. The utilized sand was washed sand so that its

TABLE 2

Physical properties of the OPC used for the tests.

Property	Unit	Value
Fineness (Blaine)	m ² /kg (ft ² /lb)	411 (2007)
Specific gravity	–	3.123
Water demand	%	28.5
Compressive strength, 1 day	MPa (lb/in. ²)	23 (3336)
Compressive strength, 2 days	MPa (lb/in. ²)	37 (5367)
Compressive strength, 7 days	MPa (lb/in. ²)	50 (7252)
Compressive strength, 28 days	MPa (lb/in. ²)	61 (8847)

TABLE 3

Mixture composition of SCCs and paste of STA.

Constituent		POW		STA	
		SCC	Paste	SCC	Paste
		Unit Weight, kg/m ³ (lb/ft ³)			
Cement	3125 (195.1)	310 (19.4)	804 (50.2)	350 (21.8)	987 (61.7)
Limestone filler	2735 (170.7)	250 (15.6)	649 (40.5)	130 (8.1)	367 (22.9)
Water	1000 (62.4)	175 (10.9)	454 (28.3)	175 (10.9)	494 (30.8)
Sand 0–4 mm	2650 (165.4)	808 (50.4)	–	848 (52.9)	–
Aggregate 4–16 mm	2600 (162.3)	791 (49.4)	–	831 (51.9)	–

contribution to the powder fraction was negligible. Quartzitic washed aggregate was used with a maximum grains size of 16 mm (0.63 in.).

The mixture compositions of the concretes as well as the observed paste calculated from the mixture composition of the stabilizing agent type can be found in **Table 3**. These concrete types were adjusted by means of differently charged PCE HRWRAs, which all consist of identical main chains. The different charge densities were achieved by varying the grafting degree as well as the number of polyethylene units in the grafts. All PCEs are commercial products and no specific information can be provided. Their charge density can thus be described only qualitatively. Their properties are given in **Table 4**. The PCEs were added in the specific amounts that generated slump flow values between 650 mm (24.6 in.) and 700 mm (27.6 in.) 30 min after mixing. See **Fig. 2**. These dosages are given in **Table 5**.

The rheological properties were determined using a Rheometer-4SCC for concrete measurements (See **Figs. 3** and **4**). By measuring the torque at varied rotational speeds, under assumption of Bingham behavior, the equipment allows to calculate a flow curve, which provides qualitative information about yield stress (G-Yield) and plastic viscosity (H-Viscosity) (**Figs. 5** and **6**). Due to the non-Newtonian behavior of the concrete and undefined shear fields in the rheometer, a sound conversion over a wide range of consistencies into fundamental units for yield stress and plastic viscosity in Pa and Pa·s, respectively, is not possible. However, due to the qualitative affinity of the measurement values (**Fig. 6**) and the fundamental values, changes in G-Yield and H-Viscosity likewise represent changes into the same direction for yield stress and plastic viscosity. The measurement profile can be taken from **Table 6**. As illustrated in **Fig. 5**, only the measurements of the last four seconds of each speed step were utilized to calculate the Bingham curve.

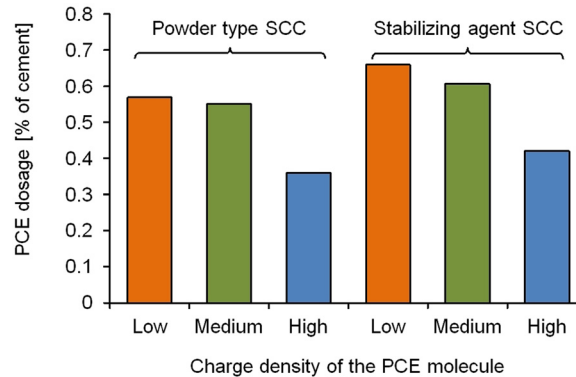
TABLE 4

Characteristics of the varied PCE.

	PCE-LC	PCE-MC	PCE-HC
Charge density (qualitative)	low	medium	High
Graft chain length	medium + low	medium + low	medium
Grafting degree	high	medium	low
Solid content	30 %	23 %	20 %

FIG. 2

Required PCE dosages to achieve slump flow diameters between 650 and 700 mm.



In order to further observe the influence of the PCE charge density beyond the workability period, investigations on the paste of the STA mixture were conducted in an automatic Vicat device (Toni Technik). The paste composition can be found in **Table 3**. In order to distinguish between the influence of the charge density of a PCE and the respective amount of PCE, two observation series were set up at varied PCE dosages. In a first series, the effective polymer contents were kept identical regardless of the effect on the flow properties. In the second series, the PCE was added in the same amount as required to achieve slump flow values in concrete between 650 mm (24.6 in.) and 700 mm (27.6 in.) 30 min after mixing. In order to observe the influence of the mixture composition, the investigation was also conducted with the high charge density PCE for the STA and the POW paste in the respective amount that was required to fulfill the specified slump flow diameter in concrete. The admixture dosages used in these tests can be taken from **Table 7**. The dosages slightly vary from those in **Table 4**, the reason for which is based in the use of a differently operating viscosity modifying agent in the paste tests than used for the concrete tests. However, the deviations in the PCE dosage between paste and concrete tests are negligibly small.

Results and Discussion

RHEOLOGICAL PROPERTIES

Analyzing the required amounts of PCE to achieve the specified flow properties gives a first indicator about the different performances of the varied PCEs (**Fig. 2**). For both mixture composition types, the addition amounts decrease with increasing

TABLE 5

HRWRA adjustment to achieve slump flow values of SCC between 650 and 700 mm after 30 min.

	PCE-LC	PCE-MC	PCE-HC
	Dosage by Weight of Cement, %		
POW	0.62	0.55	0.38
STA	0.68	0.61	0.44

FIG. 3

Concrete rheometer used for the SCC investigations.



charge density of the PCE. This confirms the initially mentioned effect of the charge density on the amount of required PCE polymers. It can also be found that for both mixture types the required amount of the low and medium charge PCEs are in the same order of magnitude, while a significantly lower amount of the high charge density polymer was required, indicating that it is likely that the low and medium charge density PCEs may behave similarly, while a distinctive performance difference can be expected with the high charge density PCE.

The results of the rheological measurements over a period of 90 min of time are shown in **Figs. 6** and **7**. Since the rheometric measurement method is sensitive to influences of the mixture composition and in particular aggregate migration, the absolute G-Yield and H-Viscosity values between POW and STA mixtures cannot be compared. However, for each mixture composition type (POW and STA), observed variations induced by HRWRA characteristics or time-dependent effects are significant.

Yield stress is typically affected by particle interactions, while viscosity is often mainly affected by changes of phase volumes. The use of differently charged PCEs

FIG. 4

Measurement program and sample electric current measurements.

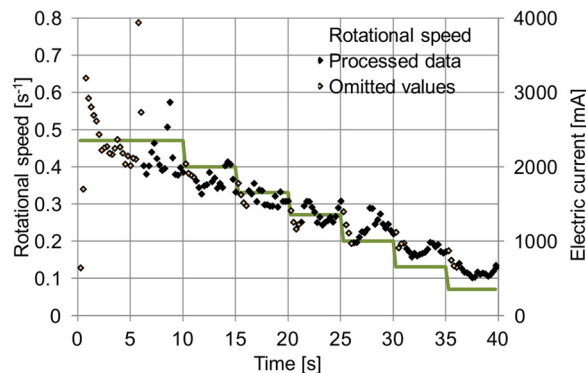


FIG. 5
Sample flow curve and
derivation of H-Viscosity and
G-Yield.

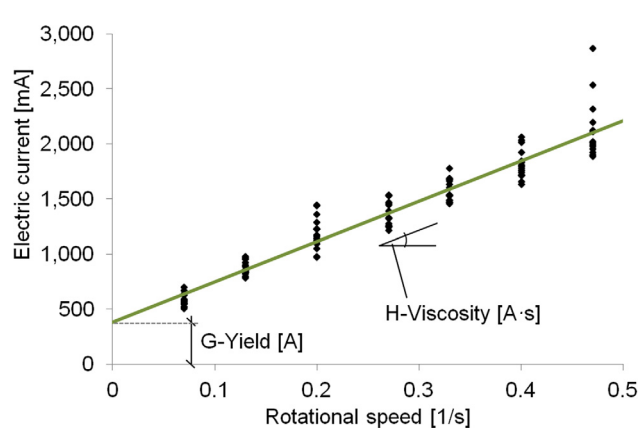


FIG. 6
H-Viscosity values of the POW
and STA mixture over a time of
90 min.

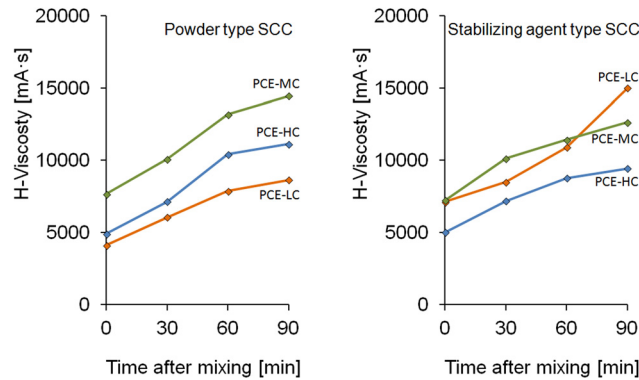


TABLE 6
Setup for rheometric concrete investigations.

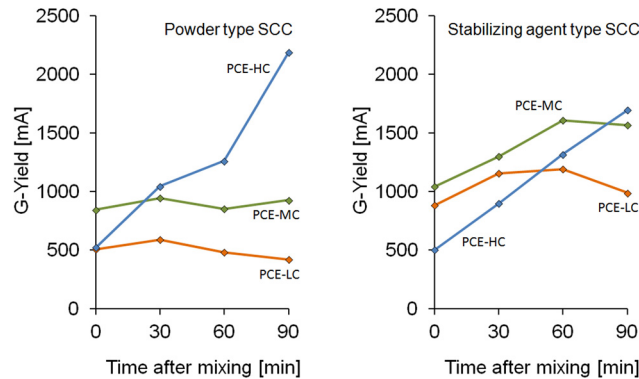
Speed, s ⁻¹	0.45	0.40	0.35	0.30	0.25	0.20	0.15	0.10	0.05
Duration, s	10	5	5	5	5	5	5	5	5

TABLE 7
Variations of the dosages and types of HRWRA in paste tests.

		PCE-LC	PCE-MC	PCE-HC
		Dosage by Weight of Cement, %		
Identical solid content	STA	0.42	0.42	0.42
Similar yield stress	STA	0.66	0.61	0.42
	POW	–	–	0.36

FIG. 7

G-Yield values of the POW and STA mixture over a time of 90 min.



therefore can be expected to mainly affect the G-Yield rather than the H-Viscosity. This is confirmed by the **Figs. 6** and **7**. Apart from an initial effect of each PCE on the H-Viscosity in general, an ongoing increasing H-Viscosity can be observed regardless of the SCC type or the charge density of the PCE (**Fig. 6**). However, the rate of increase is very similar for all PCE types. At the same time, distinct time-dependent effects can be observed for both SCC types regarding the G-Yield (**Fig. 7**).

The time resolved performance of the low and medium charge density PCEs are very similar for both mixtures, while the SCCs incorporating the high charge density PCE show a more rapid increase of the G-Yield values. This confirms the formerly introduced effect of the charge density on the time dependent flow performance of SCC. The mechanism is illustrated in **Figs. 8** and **9**. The higher charge density polymers are adsorbed rapidly onto the surfaces of aluminate and ferrous phases as well as on very early hydration products, mainly ettringite and monosulfate, while larger parts of the lower charge density polymer maintain ineffective in the solution (**Fig. 8**). However, due to the hydration, the early adsorbed high charged PCEs become ineffective by morphological changes of the particle surfaces, while the initially non-adsorbed low charge density polymers can develop a dispersing effect over a longer period of time (**Fig. 9**).

FIG. 8

Effect of PCE charge density on the dispersing ability at an early step in time.

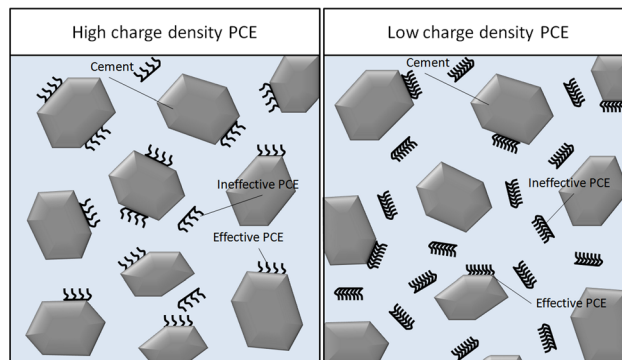
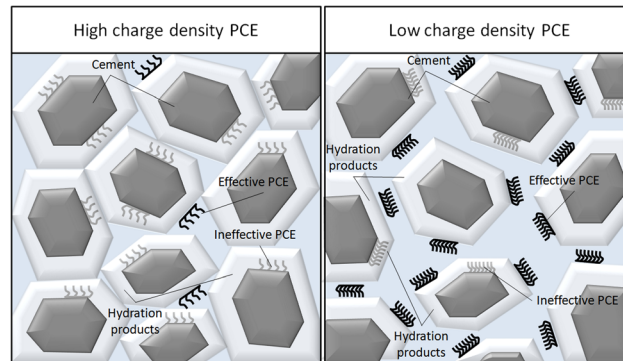


FIG. 9

Effect of PCE charge density on the dispersing ability over the course of time.



Besides the effect of the differently charged PCEs, there is a clear difference in the quality of the more rapid stiffening of the mixtures incorporating high charge density PCE. The powder type mixture shows significantly faster increase of the G-Yield. In order to understand this effect, the w/p has to be taken into account. The mode of operation of powder type SCCs is based on a dense packing of particles and narrow distances that need to be filled by the fluent phase. Stabilizing agent types have significantly larger distances between the particles and a higher volume needs to be filled up with fluid. As soon as there is no more sufficient free PCE available to effectively disperse the particles, the effect of the available water volume becomes dominant. Due to the low w/p of powder type SCCs, particles become less and less mobile among each other with ongoing hydration. The same effect occurs significantly less pronounced for stabilizing agent SCCs due to the significantly larger water volume in the entire system. This effect is illustrated in **Fig. 10**.

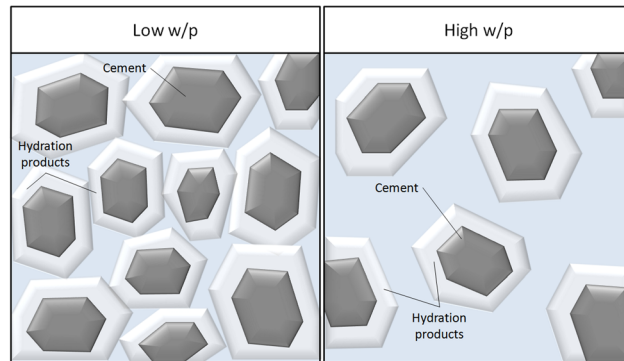
For the same reason, it is assumable that the retention of the low yield stress for the POW mixtures in case of low and medium charge PCE is better than in the STA mixtures. The ongoing dispersing effect of the time delayed adsorbing polymers may cause micro-segregation in the pastes of the STA concrete due to the high water volume. This effect also causes that stabilizing agent types perform poorly at very low temperatures, when the adsorption of PCEs is hindered due to the slow velocity of the hydration reaction [12].

SETTING AND EARLY HYDRATION

Presently, there is no clarification on why PCEs slow down the hydration. The modes of operation discussed in literature are hindrance of alite dissolution, possible calcium ion complexation, prevented C-S-H precipitation, and changed growth or nucleation of hydrates [13–16]. It is well known that increasing PCE dosages intensify hydration process retardation [17–19]. However, there is little awareness of the great effect that the charge density of a PCE on the extent of delay in the further course of hydration has. The results of the set experiments are shown in **Fig. 11**. If the three differently charged PCEs are added in identical effective agent contents (0.42 % bwoc), it can be observed that the different PCEs affect the paste setting

FIG. 10

Effect of w/b on the particle mobility over the course of time.

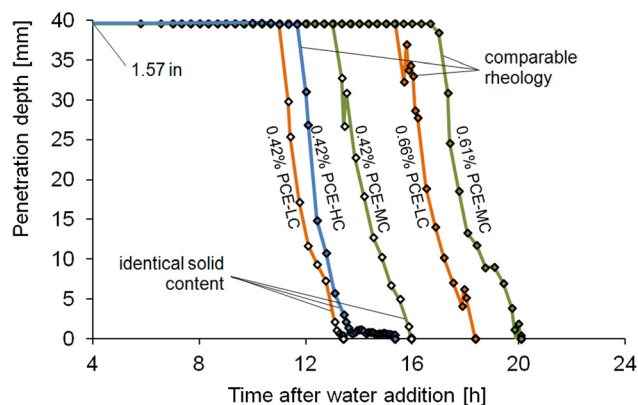


behavior in different ways. The lowest charge density PCE sets most early; however, the highest charge density PCE shows similar behavior with only a small time shift in both initial and final setting. The medium charge density PCE, however, causes a stronger set retardation. The lack of a systematic influence of the charge density of the set retardation clearly indicates that the charge density solely might not be a reasonable parameter to be linked to the mechanisms that control the set retardation.

However, any effect of the polymer setup is negligible in contrast to the dominant influence of the amount of polymers required for the specified flow properties. As shown in **Fig. 11**, if differently charged PCEs are added in the effective amounts that are required to adjust similar slump flow values (0.66, 0.61, and 0.42 % for LC, MC, and HC, respectively), the influence of the charge density can be identified. The high charge density PCE causes significantly earlier setting, while the low and medium charge density polymers cause a shift of the setting times between 3 and 4 h. The time effects thus are dominated by effects caused purely by the amount of PCE in the system. Since the high charge density PCE can be adsorbed more efficiently, significantly lower amounts are required to achieve the specified slump flow value. The low and medium charge density polymers, which require higher dosages,

FIG. 11

Effect of charge density and dosage of PCE on the setting behavior of SCC paste.



set significantly slower. Since they both are added in similar amounts, they also show similar effects on the set retardation.

For production of SCC, PCEs are usually added with focus on the rheological properties. Therefore, despite the fact that obviously the effect of the PCE geometry on the setting might be complex, the charge density is the most influencing parameter caused by the fact that obviously any polymer's effect is outweighed by the influence of the amount of PCE polymers. In this context, higher charge density PCEs show less retarding effect on the hydration than lower charge density PCEs.

The setting behavior is furthermore influenced by the mixture composition. **Figure 12** shows the setting of the pastes in case of a POW and STA type SCC with high charge density. While the high powder paste POW sets around 9 h, the STA paste sets around 13 h. One influence on the earlier setting of the POW mixture is the lower PCE dosage required to adjust similar flow properties. However, the PCE dosages are in a similar order of magnitude with 0.36 % bwoc and 0.42 % bwoc. It is therefore unlikely that the lower PCE dosage in the POW mixture can be solely responsible for the distinct effect on the setting. The filler used in the investigations is limestone filler. Limestone filler is known to accelerate setting due to providing crystallization seeds in the finest components. Furthermore, PCE can be adsorbed on limestone filler as well, thus withdrawing part of the PCE from the interaction with the cement hydration. The significantly higher amount of limestone filler in the POW can be assumed to cause the earlier setting. Finally, the lower w/p in the POW mixture also accounts responsible for the earlier setting, despite its lower cement content, since the denser particle packing fosters the formation of a stiff structure caused by hydrates.

PRACTICAL APPLICATIONS

For the practitioner on the construction site, it is important to consider aspects like haul time and casting time, which may vary from construction site to construction site and demand individual workability adjustments. The longest retardation of the workability and the setting time can be achieved by using a high w/p mixture with low charge density PCE; the shortest workability time and the earliest setting can be achieved by using a high powder content mixture with a high charge density PCE.

FIG. 12

Effect of the mixture composition on the setting behavior of SCC paste.

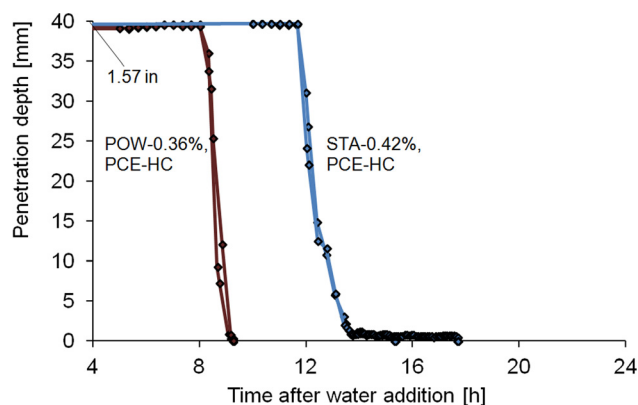
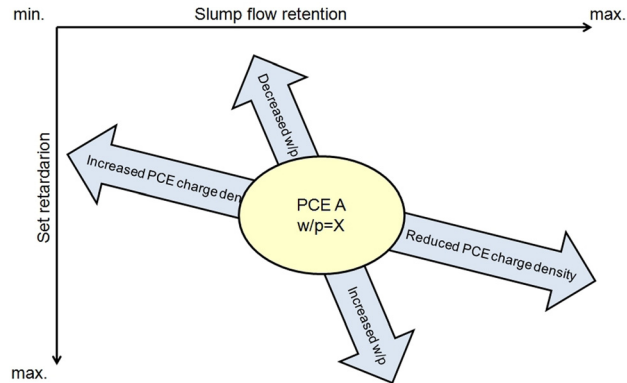


FIG. 13

Influence of the PCE and the w/p on flow retention and set retardation.



Changing any of these parameters, either charge density of the PCE or w/p of the mixture, may affect the workability and set retardation following the rule: the higher the charge density of the PCE or the lower the w/p, the shorter the slump flow retention and the earlier the setting. The observations from **Fig. 7** indicate that particularly at low w/p, the influence of the charge density of the PCE may have a strong effect on the slump flow retention, while the high powder content can be assumed to contribute positively to earlier setting. **Figure 12** indicates that in the present results, the mixture composition again has a strong influence on the set retardation, with powder rich mixes showing significantly earlier setting, while at the same time, the mixture composition influence on the retardation, as shown in **Fig. 7**, is not comparably strong. These observations are illustrated in a simplified way in **Fig. 13**. Since in the investigations ordinary Portland cement and limestone filler, which is considered to be largely inert, were used, the demonstrated effects can be assumed to have qualitative validity for a wide range of SCC mixture compositions. However, further effects may overlap.

Conclusions

Investigations on the rheological and setting behavior of two types of SCC mixtures, POW and STA, made with different w/p ratios were conducted. The influences of PCE HRWRAs with different charge density were observed. The following conclusions can be made:

For both types of SCC mixtures, the required dosage for achieving a slump flow of 650–700 mm decreases with the charge density of PCE. The required dosage for the PCE with high charge density (0.35 %–0.40 % of cement) is much lower than those for the PCEs with low and medium charge density (0.55 %–0.68 % of cement).

- The charge density of the PCE strongly affected the retention of the flow properties. For both SCC types, the low and the medium charged PCE provide the mixtures with a stable increase in H-Viscosity and a little or no increase in G-Yield, while high charge density PCE provides the mixtures with a rapid increase in both H-Viscosity and G-Yield with time.
- More rapid stiffening was observed for the powder type SCC. This can be explained by morphological changes of the particles due to hydration

reactions, which reduce the mobility of particles among each other. The denser the particles are packed initially, the stronger their negative influence on the workability retention.

- Setting times of pastes in correspondence to SCC mixtures increased with the dosage of PCE. For a given dosage, the pastes with PCE-LC set faster than the pastes with PCE-HC, which in turn set faster than the paste with PCE-MC.
- The latter effect, however, retreated into the background, when PCE was added at a dosage that was required to achieve a specified flow property. Since PCE-MC and PCE-LC were required at significantly higher dosages than PCE-HC to achieve similar rheological properties, pastes with PCE-LC and PCE-MC showed significantly more retarded setting than with PCE-HC.
- In general, effects that cause performance retention caused set retardation as well. Increasing water to powder ratios as well as decreasing charge densities of the PCE caused more extended setting times.
- The powder type SCC showed significantly earlier setting. The reason for this may be found in the lower w/p ratio, the lower PCE demand, as well as the utilization of limestone filler, which is known to accelerate the setting.

The above observation can help optimizing the casting process of SCC according to individual specifications. For short haul and casting times and a rapid construction site progress, a powder rich SCC with a high charge density can be considered as the best choice. If the casting process demands for enduring flowability retention and slow de-molding can be accepted, a high w/p mixture with low charge density should be preferred. The charge density and the mixture composition both can help finding the optimum adjustment for particular construction site conditions.

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